

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Chlorido(2-chloronicotinato)triphenylantimony(V)

Li Quan, Handong Yin* and Daqi Wang

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: handongyin@163.com

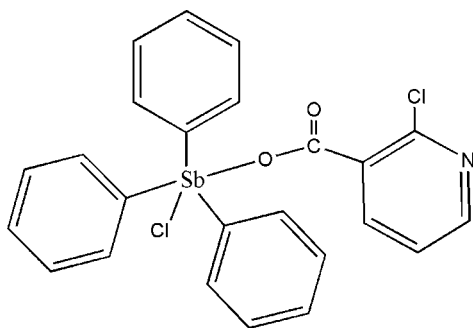
Received 9 September 2008; accepted 16 September 2008

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.024; wR factor = 0.072; data-to-parameter ratio = 14.4.

In the title complex, $[\text{Sb}(\text{C}_6\text{H}_5)_3(\text{C}_6\text{H}_3\text{ClNO}_2)\text{Cl}]$, the Sb center has a close to ideal trigonal-bipyramidal geometry, with the phenyl ligands in equatorial positions and the chloride and a carboxylate O atom in axial positions. Weak $\text{C}-\text{H}\cdots\text{O}$ contacts generate dimeric units *via* crystallographic inversion centres.

Related literature

For related structures, see: Yin *et al.* (2008); Chaudhari *et al.* (2007)



Experimental

Crystal data

$[\text{Sb}(\text{C}_6\text{H}_5)_3(\text{C}_6\text{H}_3\text{ClNO}_2)\text{Cl}]$
 $M_r = 545.04$
 Monoclinic, $P2_1/n$
 $a = 11.9580$ (9) Å
 $b = 15.4428$ (18) Å
 $c = 12.0264$ (15) Å
 $\beta = 94.291$ (2)°

$V = 2214.6$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.51$ mm⁻¹
 $T = 298$ (2) K
 $0.45 \times 0.41 \times 0.40$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.550$, $T_{\max} = 0.584$
 (expected range = 0.516–0.547)

10922 measured reflections
 3893 independent reflections
 3149 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.071$
 $S = 1.00$
 3893 reflections

271 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.52$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sb1—C19	2.107 (3)	Sb1—O1	2.114 (2)
Sb1—C13	2.111 (3)	Sb1—Cl2	2.4921 (8)
Sb1—C7	2.111 (3)		
C19—Sb1—C13	137.38 (12)	C13—Sb1—C7	108.55 (12)
C19—Sb1—C7	114.05 (12)	O1—Sb1—Cl2	177.56 (6)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C20}-\text{H20}\cdots\text{O2}^i$	0.93	2.59	3.346 (4)	139

Symmetry code: (i) $-x + 1, -y, -z + 1$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

We acknowledge the National Natural Foundation of China (grant No. 20771053) and the Natural Science Foundation of Shandong Province (2005ZX09) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2109).

References

- Chaudhari, K. R., Jain, V. K., Sagoria, V. S. & Tiekink, E. R. T. (2007). *J. Organomet. Chem.* **692**, 4928–4932.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Yin, H. D., Quan, L. & Li, L. W. (2008). *Inorg. Chem. Commun.* **11**, 1122–1125.

supplementary materials

Acta Cryst. (2008). E64, m1302 [doi:10.1107/S1600536808029632]

Chlorido(2-chloronicotinato)triphenylantimony(V)

L. Quan, H. Yin and D. Wang

Comment

The triphenylantimony(V) acetylferroceneoxime structure shows some *in vitro* antitumor activity (Yin *et al.* 2008). The title compound may show similar activities. A similar compound was synthesized (Chaudhari *et al.* 2007). The crystal structure of complex I (Fig. 1) consists of dimeric units, (Fig. 2) generated through weak intermolecular C—H...O hydrogen bonds *via* crystallographic inversion centres (Table 2). The sum of the equatorial angles C7—Sb1—C13, C13—Sb1—C19 and C19—Sb1—C7 is 359.98° and the corresponding axial angle Cl2—Sb1—O1 is 177.56 (6)° (Table 1). The distance Sb—O1 2.114 (2) Å is shorter than the range of short Sb—O distances 2.119 (3) - 2.133 (3) Å, and shorter than the range of the long Sb—O distances between 3.012 (6) and 3.112 (4) Å from related literature (Chaudhari *et al.* 2007). The Sb1—O2 distance of 2.898 (2) Å in the title complex is shorter than the sum of the van der Waals radii for Sb and O (3.2 Å), but much longer than the covalent bond Sb—O1 (2.114 (2) Å). So it can be considered that atom O2 does not make any significant contact with the Sb1 atom.

Experimental

2-chloronicotinic acid (0.044 g, 0.4 mmol) and sodium methoxide (0.8 ml, 0.4 mmol) was added to a stirring solution containing triphenylantimonydichloride (0.172 g, 0.4 mmol) in toluene (25 ml). After refluxing for 8 h, the colorless solution was obtained and then filtered. The solvent was gradually removed by evaporation under vacuum until the white solid was obtained. The solid was recrystallized from petroleum ether/dichloromethane (1:1) to give colorless crystals.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, with $U_{iso}(H) = 1.2 U_{eq}(C)$.

Figures

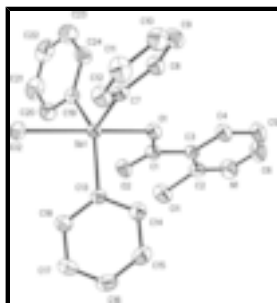


Fig. 1. The molecular structure of (I) with atom labels and 50% probability displacement ellipsoids.

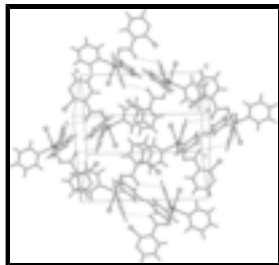


Fig. 2. A dimer linked by C20—H20...O2ⁱ hydrogen bonds (dashed lines) (symmetry code i: 1 - x, -y, 1 - z).

Chlorido(2-chloronicotinato)triphenylantimony(V)

Crystal data

[Sb(C₆H₅)₃(C₆H₃ClNO₂)Cl]

$M_r = 545.04$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.9580$ (9) Å

$b = 15.4428$ (18) Å

$c = 12.0264$ (15) Å

$\beta = 94.291$ (2)°

$V = 2214.6$ (4) Å³

$Z = 4$

$F_{000} = 1080$

$D_x = 1.635$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5771 reflections

$\theta = 2.3$ – 28.0 °

$\mu = 1.51$ mm⁻¹

$T = 298$ (2) K

Block, colorless

$0.45 \times 0.41 \times 0.40$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.550$, $T_{\max} = 0.584$

10922 measured reflections

3893 independent reflections

3149 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.2$ °

$h = -13$ → 14

$k = -17$ → 18

$l = -14$ → 10

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.071$

$S = 1.00$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.4912P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

3893 reflections

$$\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$$

271 parameters

$$\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sb1	0.523870 (16)	0.223556 (12)	0.600425 (16)	0.03369 (9)
Cl1	0.55909 (9)	-0.11472 (6)	0.80420 (9)	0.0663 (3)
Cl2	0.45726 (7)	0.27660 (6)	0.41124 (6)	0.0467 (2)
N1	0.6489 (3)	-0.0695 (2)	0.9960 (3)	0.0674 (9)
O1	0.58192 (17)	0.17341 (13)	0.75786 (17)	0.0412 (5)
O2	0.5294 (2)	0.04741 (15)	0.68384 (19)	0.0534 (6)
C1	0.5703 (2)	0.0892 (2)	0.7620 (3)	0.0389 (7)
C2	0.6110 (3)	-0.0368 (2)	0.8986 (3)	0.0472 (8)
C3	0.6124 (2)	0.0507 (2)	0.8723 (3)	0.0390 (7)
C4	0.6553 (3)	0.1057 (2)	0.9560 (3)	0.0526 (9)
H4	0.6581	0.1650	0.9431	0.063*
C5	0.6938 (3)	0.0727 (3)	1.0584 (3)	0.0671 (11)
H5	0.7220	0.1092	1.1153	0.081*
C6	0.6895 (4)	-0.0142 (3)	1.0742 (4)	0.0746 (13)
H6	0.7163	-0.0363	1.1431	0.090*
C7	0.5622 (3)	0.34849 (19)	0.6637 (3)	0.0382 (7)
C8	0.6477 (3)	0.3615 (2)	0.7465 (3)	0.0498 (9)
H8	0.6901	0.3151	0.7753	0.060*
C9	0.6690 (3)	0.4453 (2)	0.7860 (3)	0.0584 (10)
H9	0.7269	0.4549	0.8406	0.070*
C10	0.6060 (4)	0.5124 (2)	0.7453 (3)	0.0629 (11)
H10	0.6214	0.5680	0.7719	0.075*
C11	0.5199 (4)	0.4995 (2)	0.6655 (3)	0.0657 (11)
H11	0.4758	0.5459	0.6395	0.079*
C12	0.4988 (3)	0.4171 (2)	0.6237 (3)	0.0537 (9)
H12	0.4414	0.4084	0.5683	0.064*
C13	0.3560 (2)	0.1945 (2)	0.6294 (3)	0.0363 (7)
C14	0.3242 (3)	0.2102 (2)	0.7346 (3)	0.0527 (9)
H14	0.3771	0.2264	0.7914	0.063*
C15	0.2124 (3)	0.2015 (3)	0.7553 (3)	0.0649 (11)
H15	0.1895	0.2136	0.8259	0.078*
C16	0.1358 (3)	0.1752 (3)	0.6727 (3)	0.0584 (10)
H16	0.0609	0.1693	0.6872	0.070*
C17	0.1683 (3)	0.1575 (3)	0.5685 (3)	0.0612 (10)
H17	0.1158	0.1383	0.5131	0.073*
C18	0.2789 (3)	0.1682 (2)	0.5453 (3)	0.0510 (9)
H18	0.3010	0.1578	0.4740	0.061*
C19	0.6570 (3)	0.1635 (2)	0.5247 (2)	0.0388 (7)
C20	0.6410 (3)	0.0945 (2)	0.4541 (3)	0.0559 (9)
H20	0.5701	0.0701	0.4409	0.067*

supplementary materials

C21	0.7306 (4)	0.0614 (3)	0.4028 (3)	0.0727 (12)
H21	0.7202	0.0144	0.3548	0.087*
C22	0.8339 (4)	0.0967 (3)	0.4217 (4)	0.0769 (14)
H22	0.8942	0.0735	0.3874	0.092*
C23	0.8497 (3)	0.1667 (3)	0.4913 (4)	0.0723 (13)
H23	0.9206	0.1913	0.5026	0.087*
C24	0.7621 (3)	0.2011 (3)	0.5444 (3)	0.0544 (9)
H24	0.7729	0.2482	0.5922	0.065*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.03589 (13)	0.02844 (13)	0.03649 (13)	-0.00131 (9)	0.00101 (9)	0.00020 (9)
C11	0.0809 (7)	0.0349 (5)	0.0805 (7)	-0.0049 (5)	-0.0123 (5)	0.0016 (5)
C12	0.0547 (5)	0.0472 (5)	0.0381 (4)	0.0028 (4)	0.0029 (4)	0.0067 (4)
N1	0.078 (2)	0.055 (2)	0.067 (2)	0.0044 (18)	-0.0095 (18)	0.0224 (18)
O1	0.0499 (12)	0.0300 (12)	0.0424 (12)	-0.0017 (10)	-0.0044 (10)	0.0024 (10)
O2	0.0722 (16)	0.0367 (13)	0.0488 (14)	-0.0085 (12)	-0.0122 (12)	0.0001 (11)
C1	0.0393 (17)	0.0341 (18)	0.0429 (18)	-0.0006 (14)	0.0007 (14)	-0.0014 (15)
C2	0.0437 (18)	0.043 (2)	0.055 (2)	0.0016 (16)	-0.0005 (16)	0.0041 (17)
C3	0.0362 (16)	0.0378 (18)	0.0423 (18)	0.0015 (14)	-0.0006 (13)	0.0010 (14)
C4	0.061 (2)	0.045 (2)	0.050 (2)	-0.0025 (17)	-0.0085 (17)	0.0012 (17)
C5	0.077 (3)	0.071 (3)	0.049 (2)	-0.002 (2)	-0.017 (2)	-0.001 (2)
C6	0.091 (3)	0.077 (3)	0.052 (2)	0.005 (3)	-0.018 (2)	0.017 (2)
C7	0.0446 (17)	0.0273 (16)	0.0438 (18)	-0.0015 (14)	0.0103 (15)	-0.0004 (14)
C8	0.061 (2)	0.037 (2)	0.051 (2)	-0.0001 (17)	-0.0046 (17)	-0.0006 (16)
C9	0.069 (2)	0.051 (2)	0.054 (2)	-0.012 (2)	-0.0057 (19)	-0.0107 (18)
C10	0.093 (3)	0.033 (2)	0.063 (3)	-0.009 (2)	0.007 (2)	-0.0060 (18)
C11	0.092 (3)	0.036 (2)	0.068 (3)	0.012 (2)	-0.005 (2)	-0.0024 (19)
C12	0.063 (2)	0.038 (2)	0.058 (2)	0.0060 (17)	-0.0044 (18)	-0.0056 (17)
C13	0.0349 (16)	0.0318 (17)	0.0420 (18)	0.0006 (13)	0.0026 (14)	0.0061 (13)
C14	0.048 (2)	0.066 (3)	0.045 (2)	-0.0009 (18)	0.0041 (16)	-0.0008 (17)
C15	0.055 (2)	0.087 (3)	0.055 (2)	0.000 (2)	0.0178 (19)	0.001 (2)
C16	0.0376 (18)	0.065 (3)	0.074 (3)	0.0008 (18)	0.0122 (19)	0.013 (2)
C17	0.0403 (19)	0.076 (3)	0.067 (3)	-0.0089 (19)	-0.0027 (18)	-0.002 (2)
C18	0.0430 (19)	0.062 (2)	0.048 (2)	-0.0040 (18)	0.0016 (16)	-0.0052 (18)
C19	0.0426 (17)	0.0341 (17)	0.0398 (17)	0.0059 (14)	0.0038 (14)	0.0010 (14)
C20	0.069 (2)	0.041 (2)	0.058 (2)	-0.0052 (18)	0.0049 (19)	-0.0097 (17)
C21	0.101 (3)	0.054 (3)	0.065 (3)	0.023 (3)	0.024 (2)	-0.009 (2)
C22	0.075 (3)	0.090 (4)	0.068 (3)	0.042 (3)	0.024 (2)	0.011 (3)
C23	0.041 (2)	0.101 (4)	0.075 (3)	0.012 (2)	0.004 (2)	0.006 (3)
C24	0.0442 (19)	0.064 (2)	0.055 (2)	0.0044 (18)	-0.0025 (17)	-0.0053 (18)

Geometric parameters (\AA , $^\circ$)

Sb1—C19	2.107 (3)	C11—C12	1.385 (5)
Sb1—C13	2.111 (3)	C11—H11	0.9300
Sb1—C7	2.111 (3)	C12—H12	0.9300
Sb1—O1	2.114 (2)	C13—C14	1.369 (5)

Sb1—C12	2.4921 (8)	C13—C18	1.377 (4)
C11—C2	1.736 (4)	C14—C15	1.385 (5)
N1—C2	1.324 (4)	C14—H14	0.9300
N1—C6	1.335 (5)	C15—C16	1.361 (5)
O1—C1	1.309 (4)	C15—H15	0.9300
O2—C1	1.213 (4)	C16—C17	1.367 (5)
C1—C3	1.506 (4)	C16—H16	0.9300
C2—C3	1.389 (5)	C17—C18	1.382 (5)
C3—C4	1.386 (5)	C17—H17	0.9300
C4—C5	1.380 (5)	C18—H18	0.9300
C4—H4	0.9300	C19—C20	1.367 (5)
C5—C6	1.356 (6)	C19—C24	1.390 (5)
C5—H5	0.9300	C20—C21	1.374 (5)
C6—H6	0.9300	C20—H20	0.9300
C7—C12	1.369 (5)	C21—C22	1.354 (6)
C7—C8	1.388 (4)	C21—H21	0.9300
C8—C9	1.394 (5)	C22—C23	1.371 (6)
C8—H8	0.9300	C22—H22	0.9300
C9—C10	1.351 (5)	C23—C24	1.373 (5)
C9—H9	0.9300	C23—H23	0.9300
C10—C11	1.369 (5)	C24—H24	0.9300
C10—H10	0.9300		
C19—Sb1—C13	137.38 (12)	C10—C11—C12	119.7 (4)
C19—Sb1—C7	114.05 (12)	C10—C11—H11	120.1
C13—Sb1—C7	108.55 (12)	C12—C11—H11	120.1
C19—Sb1—O1	91.02 (10)	C7—C12—C11	120.2 (3)
C13—Sb1—O1	91.53 (10)	C7—C12—H12	119.9
C7—Sb1—O1	87.95 (10)	C11—C12—H12	119.9
C19—Sb1—C12	87.14 (8)	C14—C13—C18	120.9 (3)
C13—Sb1—C12	88.72 (8)	C14—C13—Sb1	116.6 (2)
C7—Sb1—C12	94.27 (9)	C18—C13—Sb1	122.3 (2)
O1—Sb1—C12	177.56 (6)	C13—C14—C15	119.2 (4)
C2—N1—C6	117.5 (3)	C13—C14—H14	120.4
C1—O1—Sb1	111.67 (19)	C15—C14—H14	120.4
O2—C1—O1	122.5 (3)	C16—C15—C14	120.2 (4)
O2—C1—C3	124.1 (3)	C16—C15—H15	119.9
O1—C1—C3	113.4 (3)	C14—C15—H15	119.9
N1—C2—C3	124.3 (3)	C15—C16—C17	120.5 (3)
N1—C2—C11	113.3 (3)	C15—C16—H16	119.8
C3—C2—C11	122.4 (3)	C17—C16—H16	119.8
C4—C3—C2	116.2 (3)	C16—C17—C18	120.2 (3)
C4—C3—C1	118.6 (3)	C16—C17—H17	119.9
C2—C3—C1	125.2 (3)	C18—C17—H17	119.9
C5—C4—C3	120.1 (4)	C13—C18—C17	119.0 (3)
C5—C4—H4	119.9	C13—C18—H18	120.5
C3—C4—H4	119.9	C17—C18—H18	120.5
C6—C5—C4	118.5 (4)	C20—C19—C24	121.0 (3)
C6—C5—H5	120.7	C20—C19—Sb1	122.5 (3)
C4—C5—H5	120.7	C24—C19—Sb1	116.4 (2)

supplementary materials

N1—C6—C5	123.3 (4)	C19—C20—C21	119.4 (4)
N1—C6—H6	118.3	C19—C20—H20	120.3
C5—C6—H6	118.3	C21—C20—H20	120.3
C12—C7—C8	119.9 (3)	C22—C21—C20	120.5 (4)
C12—C7—Sb1	118.8 (2)	C22—C21—H21	119.8
C8—C7—Sb1	121.3 (2)	C20—C21—H21	119.8
C7—C8—C9	119.0 (3)	C21—C22—C23	120.2 (4)
C7—C8—H8	120.5	C21—C22—H22	119.9
C9—C8—H8	120.5	C23—C22—H22	119.9
C10—C9—C8	120.4 (4)	C22—C23—C24	120.9 (4)
C10—C9—H9	119.8	C22—C23—H23	119.5
C8—C9—H9	119.8	C24—C23—H23	119.5
C9—C10—C11	120.8 (4)	C23—C24—C19	118.0 (4)
C9—C10—H10	119.6	C23—C24—H24	121.0
C11—C10—H10	119.6	C19—C24—H24	121.0

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C20-H20\cdots O2^i$	0.93	2.59	3.346 (4)	139

Symmetry codes: (i) $-x+1, -y, -z+1$.

Fig. 1

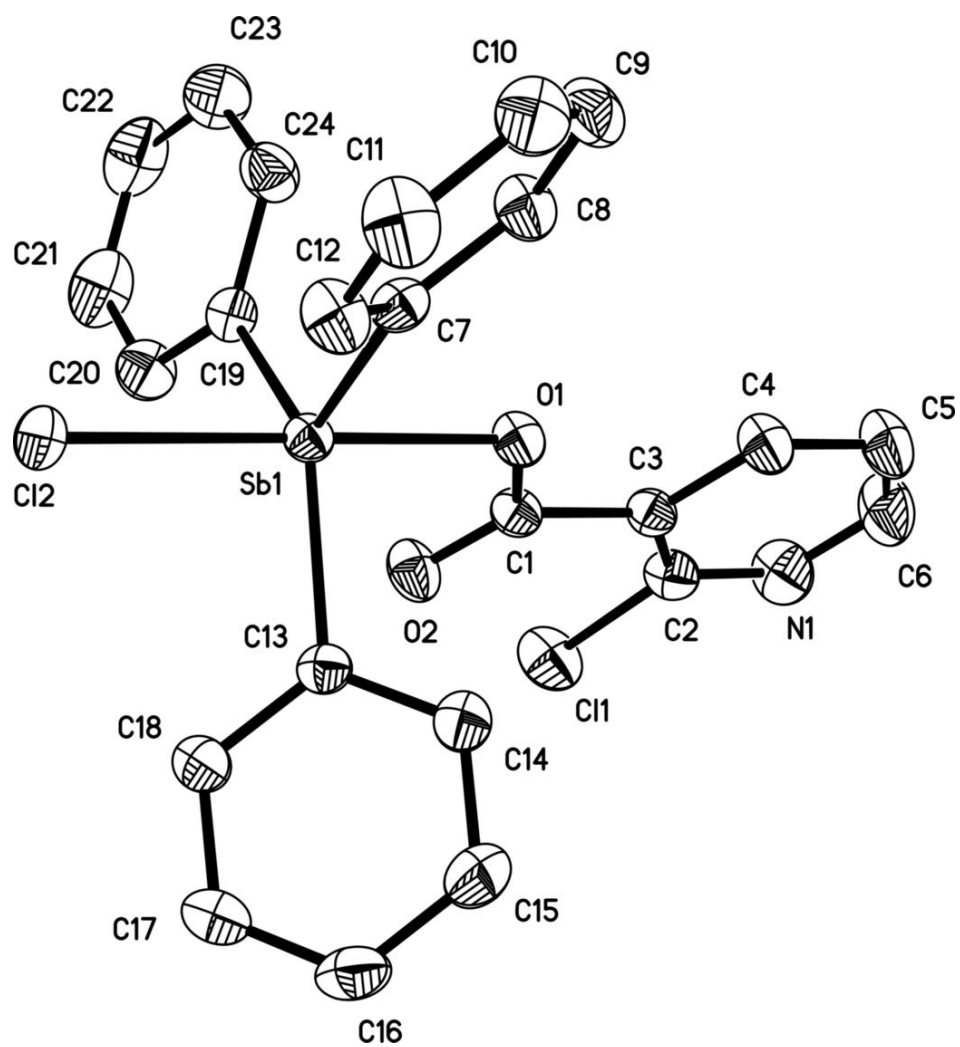


Fig. 2

